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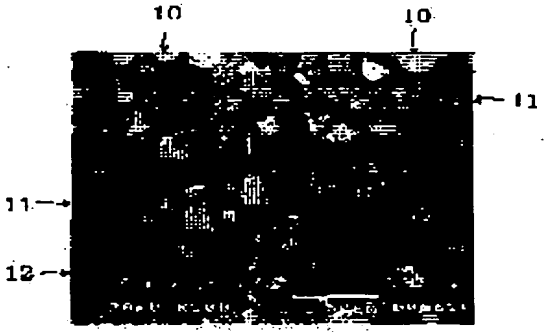
(54) HONEYCOMB STRUCTURED BODY AND METHOD OF MANUFACTURING THE SAME

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a honeycomb structured body which is sufficiently porous with high specific surface area, preferably useable for automobile exhausting gas purifying filter even under high SV condition, containing fire resistant powder such as silicon carbide but can be economically manufactured with relatively low firing temperature, and the thermal conductivity of which is set at a proper value, by processing of plugging or catalyst supporting, etc.
SOLUTION: The porous honeycomb structured body is composed of a lot of axial through holes surrounded by partitions containing aggregates of fire resistant powder 11, and metallic silicon 10.



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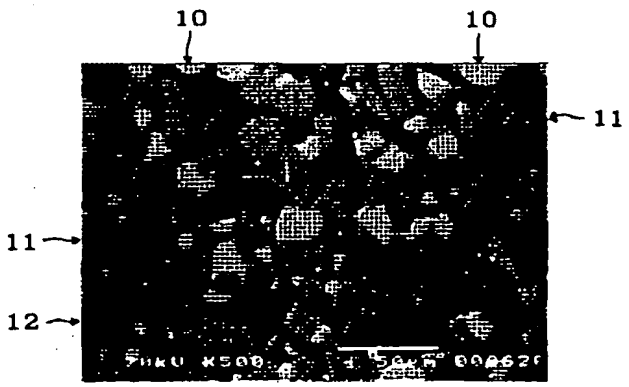
(54)【発明の名称】ハニカム構造体及びその製造方法

(57)【要約】
【課題】　炭化珪素粒子のような耐火性粒子を含みながらも比較的低い焼成温度で安価に製造できるとともに、熱伝導率が適度な数値に設定されており、十分に多孔質かつ高比表面積で、目封じや触媒担持等の処理により自動車排気ガス浄化用のフィルターとして高ＳＶ条件下でも好適に使用できるハニカム構造体を提供する。
【解決手段】　隔壁により仕切られた軸方向に貫通する多数の流通孔を有するハニカム構造体であって、骨材となる耐火性粒子１１と金属珪素１０とを含み、多孔質であるハニカム構造体である。



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【特許請求の範囲】

【請求項１】　隔壁により仕切られた軸方向に貫通する多数の流通孔を有するハニカム構造体であって、骨材となる耐火性粒子と金属珪素とを含み、多孔質であることを特徴とするハニカム構造体。

【請求項２】　前記耐火性粒子が、その耐火性粒子表面の一部において前記金属珪素により結合された構造を有する請求項１記載のハニカム構造体。

【請求項３】　熱伝導率が５Ｗ／ｍＫ以上である請求項１記載のハニカム構造体。

【請求項４】　前記耐火性粒子が、その原料粒子形状を留めた状態で前記金属珪素により結合された構造を有する請求項１記載のハニカム構造体。

【請求項５】　前記耐火性粒子が、炭化珪素粒子である請求項１記載のハニカム構造体。

【請求項６】　含塵流体中に含まれる粒子状物質を捕集除去するフィルターとして用いられる請求項１記載のハニカム構造体。

【請求項７】　気孔率が３０～９０％の範囲にある請求項１記載のハニカム構造体。

【請求項８】　平均細孔径が２～５０μｍの範囲にある請求項１記載のハニカム構造体。

【請求項９】　気孔率が５０～９０％の範囲にあるとともに、熱伝導率が５～３０Ｗ／ｍＫの範囲にある請求項２記載のハニカム構造体。

【請求項１０】　前記金属珪素の含有量が、前記耐火性粒子原料と金属珪素との合計量に対して、５～５０重量％の範囲である請求項１記載のハニカム構造体。

【請求項１１】　前記隔壁の厚さが１０２～１２７０μｍである請求項１記載のハニカム構造体。

【請求項１２】　前記隔壁の厚さとハニカム構造体の気孔率とが以下の関係を満たす請求項１記載のハニカム構造体。

【数１】隔壁の厚さ（μｍ）≧気孔率（％）×４

【請求項１３】　前記隔壁の厚さとハニカム構造体の気孔率とが以下の関係を満たす請求項１記載のハニカム構造体。

【数２】隔壁の厚さ（μｍ）≧気孔率（％）×５

【請求項１４】　前記隔壁の厚さとハニカム構造体の気孔率とが以下の関係を満たす請求項１記載のハニカム構造体。

【数３】隔壁の厚さ（μｍ）≦気孔率（％）×２０

【請求項１５】　セル密度が０．７～１５５セル／ｃｍ^{2}である請求項１記載のハニカム構造体。

【請求項１６】　耐火性粒子原料に、金属珪素と有機バインダーを添加し混合及び混練して得られた坏土をハニカム形状に成形し、得られた成形体を仮焼して成形体中の有機バインダーを除去した後、本焼成することを特徴とするハニカム構造体の製造方法。

【請求項１７】　前記耐火性粒子原料が、炭化珪素粒子原料である請求項１６記載の製造方法。

【請求項１８】　前記耐火性粒子原料の平均粒径が、最終的に得られるハニカム構造体の平均細孔径の２～４倍である請求項１６記載の製造方法。

【請求項１９】　前記金属珪素の添加量が、前記耐火性粒子原料と金属珪素との合計量に対して、５～５０重量％の範囲である請求項１６記載の製造方法。

【請求項２０】　前記金属珪素の平均粒径が、骨材である耐火性粒子の平均粒径の５０％以下である請求項１６記載の製造方法。

【請求項２１】　前記有機バインダーを、前記耐火性粒子原料と金属珪素との合計量に対して、外配で２～３０重量％の範囲で添加する請求項１６記載の製造方法。

【請求項２２】　前記坏土を調合する際に、造孔剤を、前記耐火性原料粒子と金属珪素との合計量に対して、外配で３０重量％以下の範囲で添加する請求項１６記載の製造方法。

【請求項２３】　前記成形体の仮焼を、前記金属珪素が溶融する温度より低い温度にて実施する請求項１６記載の製造方法。

【請求項２４】　前記本焼成を、１４００～１８００℃の温度範囲で実施する請求項１６記載の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】　本発明は、自動車排気ガス浄化用のフィルターや触媒担体等に使用されるハニカム構造体に関する。

【０００２】

【従来の技術】　ディーゼルエンジン排気ガスのような含塵流体中に含まれる粒子状物質を捕集除去するためのフィルター、あるいは排気ガス中の有害物質を浄化する触媒成分を担持するための触媒担体として、多孔質のハニカム構造体が広く使用されている。また、このようなハニカム構造体の構成材料として、炭化珪素（ＳｉＣ）粒子のような耐火性粒子を使用することが知られている。

【０００３】　具体的な関連技術として、例えば特開平６－１８２２２８号公報には、所定の比表面積を有するとともに不純物を含有する炭化珪素粉末を出発原料とし、これを所望の形状に成形、乾燥後、１６００～２２００℃の温度範囲で焼成して得られるハニカム構造の多孔質炭化珪素質触媒担体が開示されている。

【０００４】　一方、特開昭６１－２６５５０号公報には、易酸化性素材、又は易酸化性素材を含有する耐火組成物にガラス化素材を添加し、結合材とともに混合、混練及び成形し、成形した成形体を非酸化雰囲気の炉内で裸焼成することを特徴とするガラス化素材含有耐火物の製造方法が、特開平８－１６５１７１号公報には、炭化珪素粉末に、有機バインダーと、粘土鉱物系、ガラス系、珪酸リチウム系の無機バインダーを添加して成形す

る炭化珪素成形体が、それぞれ開示されている。

【０００５】　また、前記特開平６－１８２２２８号公報には、従来の多孔質炭化珪素質焼結体の製造方法として、骨材となる炭化系素粒子にガラス質フラックス、あるいは粘土質などの結合材を加え成形した後、その成形体を前記結合材が溶融する温度で焼き固めて製造する方法も紹介されている。

【０００６】　更に、特公昭６１－１３８４５号公報及び特公昭６１－１３８４６号公報には、珪砂、陶磁器粉砕物、Al_2O_3、TiO_2、ZrO_2等の金属酸化物、炭化珪素、窒化物、硼化物あるいはその他の耐火性材料等よりなる所定粒度に整粒された耐火性粒子が、水ガラス、フリット、釉薬等の耐火性結合材で多孔質の有底筒状体に形成された高温用セラミックフィルターについて、その好適な耐火性粒子平均径、耐火性粒子粒度分布、筒状体気孔率、筒状体平均細孔径、筒状体細孔容積、筒状体隔壁肉厚等が開示されている。

【０００７】　なお、特公平８－１３７０６号公報においては、金属珪素を介して一体に接合してなる構造を有する炭化珪素／金属珪素複合体、及び、珪素集積バイオマスをアルゴン又は窒素雰囲気下で加熱処理して形成された炭化珪素と金属珪素を用いた前記複合体の製造方法が開示されている。

【０００８】

【発明が解決しようとする課題】　前記特開平６－１８２２２８号公報に示される、炭化珪素粉末自体の再結晶反応による焼結形態（ネッキング）では、炭化珪素粒子表面から炭化珪素成分が蒸発し、これが粒子間の接触部（ネック部）に凝縮することで、ネック部が成長し結合状態が得られるが、炭化珪素を蒸発させるには、非常に高い焼成温度が必要であるため、これがコスト高を招き、かつ、熱膨張率の高い材料を高温焼成しなければならないために、焼成歩留が低下するという問題があった。

【０００９】　また、上記の炭化珪素粉末自体の再結晶反応による焼結によって、高気孔率であるフィルター、特に５０％以上の気孔率を有するフィルターを製造しようとすると、当該焼結機構が十分に機能しなくなるためにネック部の成長が妨げられ、これに起因してフィルターの強度が低下してしまうといった不具合を有していた。

【００１０】　更に、上記の材料は熱伝導率が３０Ｗ／ｍＫ以上と非常に高く、局所的な発熱を抑えるという点では有利ながら、例えば触媒を担持してパティキュレートを酸化及び燃焼し、連続的に再生する方式のフィルターに用いた場合、パティキュレートの堆積量が少なく、放熱しやすいといった特徴により、担体の温度が上がるまでに非常に時間を要する。したがって、触媒が機能する温度まで温度が上がるのに時間を要するため、パティキュレートの燃え残りが生じて再生効率が下がる等の問題も併せもっていた。

【００１１】　特開昭６１－２６５５０号公報や特開平６－１８２２２８号公報に示される、原料炭化珪素粉末をガラス質で結合させる手法は、焼成温度としては１０００～１４００℃と低くて済むが、例えばこの手法で作製された焼結体をディーゼルエンジンから排出される排気ガス中に含まれるパティキュレートを除去するためのディーゼルパティキュレートフィルター（ＤＰＦ）の材料として用いる場合には、フィルター再生のため、フィルターに捕集され堆積したパティキュレートを燃焼させようとすると、熱伝導率が小さいために局所的な発熱が生じるという問題点があった。

【００１２】　更に、特公昭６１－１３８４５号公報及び特公昭６１－１３８４６号公報に示されるフィルターは、多孔質ではあるものの、隔壁が５～２０ｍｍと厚い有底筒状体であり、自動車排気ガス浄化用フィルターのような高ＳＶ（空間速度）条件下には適用できなかった。

【００１３】　また、特公平８－１３７０６号公報に示される複合体、及びその製造方法では、当該複合体を多孔質とすることもできるが、フィルターとして使用するに際しては十分な気孔率を確保することが容易ではなく、特に当該複合体をディーゼルエンジン排気ガスのような含塵流体中に含まれる粒子状物質を捕集除去するためのフィルターとして使用することは困難であった。

【００１４】　本発明は、このような従来の事情に鑑みてなされたものであり、炭化珪素粒子のような耐火性粒子を含みながらも比較的低い焼成温度で安価に製造できるとともに、熱伝導率が適度な数値に設定されており、十分に多孔質かつ高比表面積で、目封じや触媒担持等の処理により自動車排気ガス浄化用のフィルターとして高ＳＶ条件下でも好適に使用できるハニカム構造体とその製造方法を提供することを目的とする。

【００１５】

【課題を解決するための手段】　本発明によれば、隔壁により仕切られた軸方向に貫通する多数の流通孔を有するハニカム構造体であって、骨材となる耐火性粒子と金属珪素とを含み、多孔質であることを特徴とするハニカム構造体、が提供される。

【００１６】　また、本発明によれば、耐火性粒子原料に、金属珪素と有機バインダーを添加し混合及び混練して得られた坏土をハニカム形状に成形し、得られた成形体を仮焼して成形体中の有機バインダーを除去した後、本焼成することを特徴とするハニカム構造体の製造方法、が提供される。

【００１７】

【発明の実施の形態】　前記のとおり、本発明のハニカム構造体は、耐火性粒子とともにそれら耐火性粒子を結合するための金属珪素を含んでいるので、その製造時において比較的低い焼成温度で焼結させることができ、製

造コストを抑えるとともに歩留まりを向上させることができる。また、耐火性粒子の結合に金属珪素を利用したことにより、耐火性粒子の結合にガラス質を利用した従来の構造体に比して高い熱伝導率を有するので、例えばＤＰＦに使用した場合において、フィルター再生のために堆積したパティキュレートを燃焼させても、フィルターを損傷させるような局所的な発熱が生じない。更に、本発明は、特公昭６１－１３８４５号公報や特公昭６１－１３８４６号公報に示されるような厚壁の有底筒状体ではなく、多孔質のハニカム構造体であるので、自動車排気ガス浄化用のフィルターや触媒担体等として高ＳＶ条件下で使用できる。

【００１８】　また、本発明のハニカム構造体は、当該ハニカム構造体を構成する耐火性粒子が、その粒子表面の一部において金属珪素により結合された構造を有することが好ましい。図２に、本発明に係るハニカム構造体であって、炭化珪素質焼結体の結晶構造である顕微鏡写真を示す。図中、白色部分が金属珪素１０、灰色部分が炭化珪素粒子１１、黒色部分が気孔１２である。このように、耐火性粒子である炭化珪素粒子１１はその粒子表面の一部において、周囲に存在する粒子同士が金属珪素１０により結合されていることがわかる。なお、図２に示す炭化珪素質焼結体の製造方法は後述する。

【００１９】　上記構造は必要以上の金属珪素を用いることなく形成されるため、焼成の過程において生起される金属珪素同志の融合による緻密化を抑えることができる。このため、フィルターとして用いた場合の圧力損失を低く抑えるのに、十分な気孔率が確保されている。さらには、高い熱伝導率をも有しているために、したがって、例えばディーゼルエンジンから排出される排気ガス中に含まれるパティキュレートを捕集除去するためのＤＰＦ等として用いる場合における高気孔率が十分に確保されるとともに、フィルター再生のために堆積したパティキュレートを燃焼させても、高い熱伝導率を有しているために、フィルターが損傷するような局所的な発熱が生ずることはない。

【００２０】　本発明のハニカム構造体は、前記のような局所的な発熱を回避する観点から、その熱伝導率が５Ｗ／ｍＫ以上であることが好ましい。

【００２１】　また、本発明のハニカム構造体は、その微構造として、耐火性粒子が、その原料粒子形状を留めた状態で金属珪素により結合された構造を有することが好ましい。本発明のハニカム構造体を、含塵流体中に含まれる粒子状物質を捕集除去するためのフィルターとして用いる場合には、その気孔率を３０～９０％の範囲とすることが好ましい。ハニカム構造体の気孔率が３０％未満では濾過速度が不足し、９０％を超えると構造体としての強度が不足する。更に、自動車排気ガス浄化用フィルター等の圧力損失が懸念される用途に用いる場合には、気孔率を４０％以上とすることが好ましい。

【００２２】　更に、触媒を担持してパティキュレートを連続して燃焼させる方式のフィルター等、圧力損失を低く抑えなければならないフィルターとして用いるハニカム構造体である場合には、気孔率が５０～９０％、熱伝導率が５～３０Ｗ／ｍＫの範囲にあることが好ましく、気孔率が５０～８０％、熱伝導率が７～２８Ｗ／ｍＫの範囲にあることが更に好ましく、気孔率が５３～７０％、熱伝導率が９～２５Ｗ／ｍＫの範囲にあることが特に好ましい。

【００２３】　触媒を担持させる方式のフィルターとして用いるハニカム構造体においては、触媒を担持することで圧力損失が上昇するため、気孔率を予め高く設定しておく必要がある。したがって、気孔率が５０％未満では、本方式のフィルターでは圧力損失が大きくなるために好ましくない。一方、気孔率が９０％を超えると、構造体としての強度が不足するために好ましくない。

【００２４】　更に、前記方式のフィルターとして用いるハニカム構造体においては、局所的な発熱による不均一な温度分布の発生によりフィルターに局所的な応力が発生するのを抑える必要がある。したがって、熱伝導率が５Ｗ／ｍＫ未満では、局所的な発熱を効果的に抑えることが困難となる。一方、熱伝導率が３０Ｗ／ｍＫを超えると放熱の効果が大きいこと、及びパティキュレートの堆積量が少ないこと等に起因し、温度が上がりにくく触媒が機能する温度にまで昇温させるのに多大な時間を要するとともに、パティキュレートの燃え残りが生じてフィルターの再生効率が下がることがあるために好ましくない。

【００２５】　なお、本発明でいうフィルターに担持される触媒とは、パティキュレートの酸化燃焼及びNO_xの分解を目的として用いられる触媒であって、具体的には白金、パラジウム、ロジウム、イリジウム、銀などの貴金属あるいはアルミナ、ジルコニア、チタニア、セリア、酸化鉄などの酸化物等を用いることができるが、本発明はこれらのものに限定されることはない。

【００２６】　同様に本発明のハニカム構造体をフィルターとして用いる場合、ハニカム構造体の平均細孔径は、濾過する対象に応じて決定することが好ましい。例えば、ディーゼルエンジンから排出される排気ガス中に含まれるパティキュレートを捕集除去するためのＤＰＦとして用いる場合には、平均細孔径を２～５０μｍの範囲とすることが好ましい。平均細孔径が２μｍ未満ではパティキュレートの少量堆積によっても著しく圧損が上昇し、逆に、５０μｍを超えるとパティキュレートの素抜けが起こるため、好ましくない。

【００２７】　本発明のハニカム構造体における金属珪素の適切な含有量は、耐火性粒子の粒径や粒子形状によっても変わるが、耐火性粒子と金属珪素の合計量に対して５～５０重量％の範囲内とすることが好ましく、１５～４０重量％の範囲内とすることが更に好ましい。５重

量％未満では、結合材が不足であるために隣接する耐火性粒子同士の金属珪素による結合が不十分であり、熱伝導率が低下するだけでなく、ハニカム構造のような薄壁の構造体を維持し得る強度を得ることが困難となる。逆に５０重量％を超えると、適切に耐火性粒子同士を結合し得る以上に金属珪素が存在することに起因して、ハニカム構造体（焼結体）が焼結により過度に収縮してしまい、気孔率低下、平均細孔径縮小などの弊害が併発してくる点において好ましくない。

【００２８】　ハニカム構造体の流通孔（セル）を仕切る隔壁の厚さは、４ｍｉｌ以上（１０２μｍ以上）とすることが好ましい。隔壁の厚さが４ｍｉｌ（１０２μｍ）未満では、構造体としての強度が不十分である。また、強度は気孔率と密接な関係にあり、本発明のハニカム構造体の場合、隔壁の厚さと気孔率とが以下の関係を満たすように隔壁の厚さを設定すれば、必要な強度が得られ、好ましいことが判明した。

【数４】隔壁の厚さ(μｍ)≧気孔率(％)×４

【００２９】　更に、隔壁の厚さと気孔率とが以下の関係を満たすように隔壁の厚さを設定すれば、十分な強度が得られるため、より好ましい。

【数５】隔壁の厚さ(μｍ)≧気孔率(％)×５

【００３０】　一方で、ＤＰＦ等のフィルターとして用いる場合には、隔壁の厚さを、５０ｍｉｌ以下（１２７０μｍ以下）とすることが好ましい。隔壁の厚さが５０ｍｉｌ（１２７０μｍ）を超えると、濾過速度不足や圧損上昇が懸念されるためである。なお、これについても気孔率と密接な関係があり、隔壁の厚さと気孔率とが以下の関係を満たすように隔壁の厚さを設定することによって、問題を回避することができる。

【数６】隔壁の厚さ(μｍ)≦気孔率(％)×２０

【００３１】　ハニカム構造体のセル密度は、５～１０００セル／平方インチ（０．７～１５５セル／ｃｍ²）の範囲とすることが好ましい。セル密度が５セル／平方インチ（０．７セル／ｃｍ²）未満では、ハニカム構造体として強度不足となるとともに、フィルターとして用いた場合には、濾過面積も不足する。逆に、１０００セル／平方インチ（１５５セル／ｃｍ²）を超えると圧損上昇を招くため、好ましくない。

【００３２】　次に、本発明のハニカム構造体の製造方法について説明する。本発明のハニカム構造体を製造するにあたっては、まず、耐火性粒子原料に金属珪素と有機バインダーとを添加して混合及び混練し、成形用の坏土を得る。

【００３３】　使用する耐火性粒子の種類は特に限定されないが、酸化物系ではAl_2O_3、ZrO_2、Y_2O_3、炭化物系ではＳｉＣ、窒化物系ではSi_3N_4、ＡｌＮ、その他ムライト等の粒子が好適に用いられ、例えば、蓄積パティキュレートの燃焼処理時にしばしば高温に晒されるＤＰＦ等の用途には、ＳｉＣ等が耐熱性が高く、好適に用いられる。なお、耐火性粒子や金属珪素に用いる原料には、Ｆｅ、Ａｌ、Ｃａなどの微量の不純物を含有するケースがあるが、そのまま使用してもよく、薬品洗浄などの化学的な処理を施して精製したものを用いてもよい。

【００３４】　耐火性粒子原料の平均粒径は、本製造方法にて最終的に得られるハニカム構造体（焼結体）の平均細孔径の２～４倍であることが好ましい。本製造方法で得られるハニカム構造体は、焼成温度が比較的低いために耐火性粒子原料の粒子形状や粒径が概ね焼成後まで維持される。したがって、前記比率が２倍未満であると、所望の細孔径に対して粒径が小さ過ぎ、結果的に、小さな耐火性粒子群が金属珪素で細長く結合されて大きな細孔を形成することになり、ハニカム構造体のような薄壁の構造体を維持し得る程高い強度を得ることができない。

【００３５】　また、例えば耐火性粒子がＳｉＣ粒子の場合、従来多孔質ハニカム構造体に適用されてきた再結晶ＳｉＣが、その反応機構から、所望とする細孔径とほぼ同等の骨材原料粒径を必要とするのに対し、本発明のハニカム構造体のように金属珪素により結合されたＳｉＣ粒子は、粒径が細孔径の２倍以上でよいので、同じ細孔径を得ようとしたときに、再結晶ＳｉＣに比べて粗い、即ち安価な原料を使用することができ、コストメリットも大きい。

【００３６】　逆に、前記比率が４倍を超える場合には、所望の細孔径に対して用いる耐火性粒子の粒径が大き過ぎ、成形の段階で耐火性粒子を密に充填することによっても、その間隙に所望の細孔を得ることが困難となり、更にフィルター用途では、気孔率低下を招く点でも好ましくない。

【００３７】　金属珪素は焼成中に溶けて耐火性粒子の表面を濡らし、粒子同士を結合する役割を担う。その適切な添加量は、耐火性粒子の粒径や粒子形状によっても変わるが、耐火性粒子と金属珪素の合計量に対して５～５０重量％の範囲内となるようにすることが好ましい。５重量％未満では、結合材が不足して、ハニカム構造のような薄壁の構造体を維持し得る強度を得ることができず、逆に５０重量％を超えると、適切に耐火性粒子同士を結合し得る以上に過剰に金属珪素が存在するため、気孔率低下、平均細孔径縮小などの弊害が併発してくる。

【００３８】　金属珪素の平均粒径は、骨材である耐火性粒子の平均粒径の５０％以下であることが好ましい。金属珪素は焼成で溶けて集合しながら耐火性粒子にまとわりつくように移動するため、その粒径が耐火性粒子の粒径の５０％を超えると、成形時に同金属珪素粒子が占有していた空間が大きな空隙となって残り、強度低下を招いたり、フィルターとして使用する場合にはフィルター効率低下（濾過漏れ）の原因となったりする。

【００３９】　また、一般に、ハニカム構造体の押出成

形時には、粒度差のある原料粉末２種以上を混合する方が滑らかに押し出すことができ、多孔体として適切な組織を得るためにも、金属珪素の平均粒径を、骨材である耐火性粒子の平均粒径の５０％以下にすることが好ましい。

【００４０】　耐火性粒子を骨材とし、金属珪素及び必要に応じて造孔剤等を配合してなる坏土を、ハニカム形状に滑らかに押出成形するため、成形助剤として、１種以上の有機バインダーを、主原料（耐火性粒子原料と金属珪素）の合計量に対し外配で２重量％以上添加することが好ましい。しかしながら、３０重量％を超える添加は、仮焼後に過剰な高気孔率を招き、強度不足に至らしめるため好ましくない。

【００４１】　更に、隔壁の厚さが２０ｍｉｌ（５０８μｍ）以下のハニカム構造体に押出成形する場合には、４～２０重量％の範囲で添加することが好ましい。添加量が４重量％未満では斯様な薄壁に押し出すことが難しく、逆に、２０重量％を超えると、押し出し後にその形状を維持することが困難となる。

【００４２】　ハニカム構造体をフィルターとして使用する場合には、気孔率を高める目的で、坏土の調合時に造孔剤を添加してもよい。造孔剤の添加量は、主原料（耐火性粒子原料と金属珪素）の合計量に対し、外配で３０重量％以下とすることが好ましい。添加量が３０重量％を超えると、過度に気孔率が高くなり強度不足に至る。

【００４３】　なお５０％以上の高気孔率であるハニカム構造体を得る場合においても、造孔剤を添加することが好ましい。このとき使用する造孔剤の種類、及び平均粒径等を適宜選択することにより、細孔径分布が制御された高気孔率であるハニカム構造体を作製することができる。即ち、本発明においては骨材である耐火性粒子の粒子間の間隙が気孔となるが、骨材である耐火性粒子の平均粒径の１．２～４倍の粒径を有する造孔剤を適当量添加することにより、耐火性粒子の粒子間の間隙と、造孔剤の焼失跡との２つの細孔径分布からなる細孔径分布を有する高気孔率のハニカム構造体を作製することができる。したがって、耐火性粒子及び造孔剤の粒径を適当に選択することで、必要な細孔径分布に対応した柔軟な材料設計が可能となる。

【００４４】　一方、細孔径が大きいハニカム構造体を作製するために、粒径の大きな耐火性粒子や金属珪素を用いる場合においても、耐火性粒子の平均粒径の０．５倍以下の粒径を有する造孔剤を適当量添加することにより、押出成形時に滑らかに坏土を押し出すことができる。したがって、成形性を下げることなく高気孔率のハニカム構造体を作製することができる。

【００４５】　使用する造孔剤の種類は、特に限定されることはないが、具体的にはグラファイト、小麦粉、澱粉、フェノール樹脂、ポリメタクリル酸メチル、ポリエチレン、ポリエチレンテレフタレート等を挙げることができる。造孔剤は、目的に応じて１種又は２種以上組み合わせて用いてもよい。

【００４６】　前記原料を常法により混合及び混練して得られた坏土を、押出成形法等により所望のハニカム形状に成形する。次いで、得られた成形体を仮焼して成形体中に含まれる有機バインダーを除去（脱脂）した後、本焼成を行う。仮焼は、金属珪素が溶融する温度より低い温度にて実施することが好ましい。具体的には、１５０～７００℃程度の所定の温度で一旦保持してもよく、また、所定温度域で昇温速度を５０℃／ｈｒ以下に遅くして仮焼してもよい。

【００４７】　所定の温度で一旦保持する手法については、使用した有機バインダーの種類と量により、一温度水準のみの保持でも複数温度水準での保持でもよく、更に複数温度水準で保持する場合には、互いに保持時間を同じにしても異ならせてもよい。また、昇温速度を遅くする手法についても同様に、ある一温度区域間のみ遅くしても複数区間で遅くしてもよく、更に複数区間の場合には、互いに速度を同じとしても異ならせてもよい。

【００４８】　仮焼の雰囲気については、酸化雰囲気でもよいが、成形体中に有機バインダーが多く含まれる場合には、仮焼中にそれ等が酸素で激しく燃焼して成形体温度を急激に上昇せしめることがあるため、N_2、Ａｒ等の不活性雰囲気で行うことによって、成形体の異常昇温を抑制することも好ましい手法である。この異常昇温の抑制は、熱膨張係数の大きい（熱衝撃に弱い）原料を用いた場合に重要な制御である。有機バインダーを、例えば主原料に対して２０重量％（外配）以上添加した場合には、前記不活性雰囲気にて仮焼するのが好ましい。また、耐火性粒子がＳｉＣ粒子である場合の他、高温での酸化が懸念されるものである場合にも、少なくとも酸化が始まる温度以上では、前記のような不活性雰囲気で仮焼を行うことによって、成形体の酸化を抑制することが好ましい。

【００４９】　仮焼とそれに続く本焼成は、同一のあるいは別個の炉にて、別工程として行ってもよく、また、同一炉での連続工程としてもよい。仮焼と本焼成を異なる雰囲気にて実施する場合には前者も好ましい手法であるが、総焼成時間、炉の運転コスト等の見地からは後者の手法も好ましい。

【００５０】　耐火性粒子が金属珪素で結合された組織を得るためには、金属珪素が軟化する必要がある。金属珪素の融点は１４１０℃であるので、本焼成の際の焼成温度は１４００℃以上とすることが好ましい。更に最適な焼成温度は、微構造や特性値から決定される。ただし、１８００℃を超える温度では金属珪素の蒸発が進んで金属珪素を介した結合が困難になるため、焼成温度としては１４００～１８００℃が適当である。

【００５１】　なお、前記の特開平６－１８２２２８号

公報に示される再結晶法を用いた製造方法は、炭化珪素粒子同士で結合するために高い熱伝導率の焼結体が得られるが、先に述べたように蒸発凝縮という機構で焼結するので、炭化珪素を蒸発させるために、本発明の製造方法よりも高い焼成温度を必要とし、実用上使用可能な炭化珪素焼結体を得るためには少なくとも１８００℃以上、通常は２０００℃以上の高温で焼成する必要がある。

【００５２】　本焼成の雰囲気については、耐火性粒子の種類によって選択することが好ましく、例えば、ＳｉＣをはじめとする炭化物の粒子、Si_3N_4、ＡｌＮに代表される窒化物の粒子等、高温での酸化が懸念されるものについては、少なくとも酸化が始まる温度以上の温度域においては、N_2、Ａｒ等の非酸化雰囲気とすることが好ましい。

【００５３】

【実施例】　以下、本発明を実施例に基づいて更に詳細に説明するが、本発明はこれらの実施例に限定されるものではない。

【００５４】（実施例１～１３、比較例１～２）表１に示すような平均粒径を有するＳｉＣ原料粉末と、平均粒径４μｍの金属Ｓｉ粉末とを、同表に示す組成となるように配合し、この粉末１００重量部に対して、有機バインダーとしてメチルセルロース６重量部、界面活性剤２．５重量部、及び水２４重量部を加え、均一に混合及び混練して成形用の坏土を得た。得られた坏土を、押出成形機にて外径４５ｍｍ、長さ１２０ｍｍ、隔壁厚さ０．４３ｍｍ、セル密度１００セル／平方インチ（１６セル／cm^2）のハニカム形状に成形した。このハニカム成形体を酸化雰囲気において５５０℃で３時間、脱脂のための仮焼を行った後、非酸化雰囲気において表１に示す焼成温度にて２時間の焼成を行い、多孔質でハニカム構造の炭化珪素焼結体を作製した。この焼結体について、水銀ポロシメーターにて平均細孔径と気孔率を、また、レーザーフラッシュ法にて熱伝導率をそれぞれ測定し、更に４点曲げ強度を測定して、その結果を表１に示した。また図２に、実施例１において作製した炭化珪素質焼結体の結晶構造である顕微鏡写真を示した。更に、配合した金属Ｓｉ粉末の量（ｗｔ％）に対して気孔率（％）、強度（ＭＰａ）、熱伝導率（Ｗ／ｍＫ）をプロットしたグラフを図１に示した。なお、Ｘ線回折にて結晶相を同定したところ、ＳｉＣ及びＳｉからなっていることが確認された。

【００５５】

【表１】

	SiC粉末の平均粒径（μｍ）	SiC粉末の配合量（wt%）	金属Si粉末の平均粒径（μｍ）	金属Si粉末の配合量（wt%）	焼成温度（℃）	平均細孔径（μｍ）	気孔率（%）	４点曲げ強度（MPa）	熱伝導率（W/mK）
実施例１	32.6	80	4	20	1450	9.0	49.0	20	21
実施例２	32.6	80	4	20	1600	10.0	44.0	25	20
実施例３	32.6	65	4	35	1450	12.0	45.0	25	25
実施例４	32.6	65	4	35	1600	13.0	42.0	28	26
実施例５	50.0	80	4	20	1450	11.6	45.0	20	21
実施例６	50.0	80	4	20	1600	13.5	49.0	22	20
実施例７	32.6	90	4	10	1450	9.0	45.0	16	15
実施例８	32.6	85	4	15	1450	9.0	47.0	20	20
実施例９	32.6	80	12	20	1450	11.0	43.0	20	20
実施例１０	32.6	80	30	20	1450	13.0	42.0	18	25
実施例１１	32.6	70	4	30	1450	12.0	47.0	27	23
実施例１２	32.6	60	4	40	1450	12.0	43.0	23	28
実施例１３	32.6	55	4	45	1450	14.0	40.0	20	30
比較例１	32.6	97	4	3	1450	8.0	45.0	3	3
比較例２	32.6	45	4	55	1450	16.0	25.0	18	23

【００５６】（考察）比較例１においては強度・熱伝導率の低下、比較例２においては気孔率の低下を確認することができた。これに対し、本発明に係る実施例１～１３においては例えば、ディーゼルエンジンから排出される排気ガス中に含まれるパティキュレートを捕集除去するためのＤＰＦ等として用いる場合に要求される気孔率・強度・熱伝導率について、十分な数値を示している。また、図１に示すグラフから、金属Ｓｉ粉末の好適な添加量は、ＳｉＣ原料粉末と金属Ｓｉ粉末の合計量に対して５～５０重量％の範囲に存在することがわかる。このことにより、本発明の優れた効果を確認することができた。

【００５７】（実施例１４～２０）表２に示すような平均粒径を有するＳｉＣ原料粉末、金属Ｓｉ粉末を、同表の組成となるように配合し、さらにこの粉末１００重量部に対して、造孔剤としてポリメタクリル酸メチルの粉末を同表に示す重量部、有機バインダーとしてメチルセルロース８重量部、界面活性剤２．５重量部、及び水２８重量部を加え、その後は実施例１～１３と同様の方法にてハニカム構造の炭化珪素焼結体を作製した。なお焼

成温度はいずれも１４５０℃にて行った。この焼結体について、水銀ポロシメーターにより平均細孔径と気孔率を、またレーザーフラッシュ法にて熱伝導率をそれぞれ測定した。

【００５８】

【表２】

	SiC粉末の平均粒径（μｍ）	SiC粉末の配合量（ｗｔ％）	金属Si粉末の平均粒径（μｍ）	金属Si粉末の配合量（ｗｔ％）	造孔剤の平均粒径（μｍ）	造孔剤の配合量（％）	気孔率（％）	平均細孔径（μｍ）	熱伝導率（Ｗ／ｍＫ）
実施例１４	32.6	80	4	20	60	20	58.0	21.0	14
実施例１５	32.6	75	4	25	12	14	53.0	13.0	25
実施例１６	47.0	85	12	15	12	20	60.0	18.0	12
実施例１７	47.0	80	12	20	12	20	58.0	15.0	16
実施例１８	68.0	85	12	15	30	20	55.0	30.0	18
実施例１９	68.0	90	12	10	60	25	66.0	40.0	10
実施例２０	32.6	80	4	20	60	30	70.0	25.0	9

【００５９】（考察）表２から明らかなように、本発明のハニカム構造体は、例えば、触媒を担持させた自動車排気ガス浄化用のフィルター等として用いる場合に要求される気孔率、熱伝導率及び平均細孔径について、十分な数値を示している。また、骨材であるＳｉＣ粉末の粒径を大きくした場合（実施例１６～１９）であっても、造孔剤の粒径や配合量を調整することによって、成形不良を起こすことなくハニカム構造体を作製することができた。このことにより、本発明の優れた効果を確認することができた。

【００６０】

【発明の効果】　以上説明したように、本発明のハニカム構造体は、炭化珪素粒子等の耐火性粒子を含みながらも、その製造時において比較的低い焼成温度で焼結させることができるので、製造コストを抑えるとともに歩留まりも向上し、安価に提供することができる。また、ガラス質を利用して耐火性粒子を結合させた従来の構造体に比して高い熱伝導率を有するので、例えばＤＰＦに使用した場合において、フィルター再生のために堆積したパティキュレートを燃焼させても、フィルターを損傷させるような局所的な発熱が生じない。更に、気孔率や熱伝導率が所定の数値範囲であり、圧力損失の低い多孔質のハニカム構造体であるので、触媒を担持させた自動車排気ガス浄化用のフィルター等として高ＳＶ条件下でも好適に使用できる。

【図面の簡単な説明】

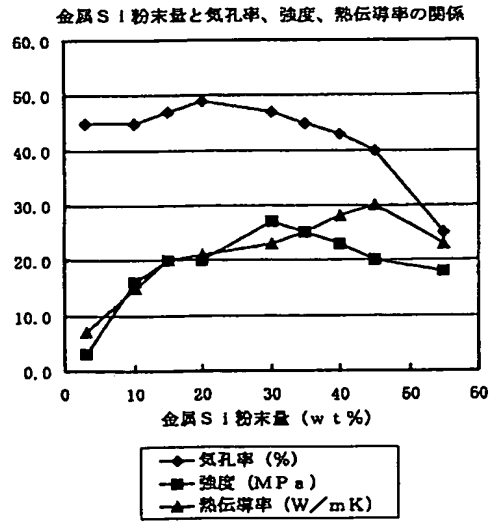
【図１】　配合した金属Ｓｉ粉末の量（ｗｔ％）に対して気孔率（％）、強度（ＭＰａ）、熱伝導率（Ｗ／ｍＫ）をプロットしたグラフである。

【図２】　実施例１において作製した炭化珪素質焼結体の結晶構造である顕微鏡写真である。

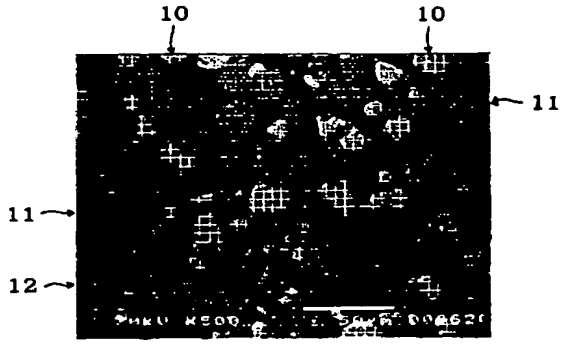
【符号の説明】

１０…金属珪素、１１…炭化珪素粒子、１２…気孔。

【図１】



【図２】



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* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the honeycomb structure object used for a filter, catalyst support, etc. for motor exhaust purification.
[0002]
[Description of the Prior Art] The porous honeycomb structure object is widely used as catalyst support for supporting the catalyst component which purifies the filter for carrying out uptake removal of the particulate matter contained in dust-containing fluid like diesel-power-plant exhaust gas, or the harmful matter in exhaust gas. Moreover, using a fireproof particle like a silicon carbide (SiC) particle as a component of such a honeycomb structure object is known.
[0003] As a concrete related technique, while having a predetermined specific surface area, the silicon carbide powder containing an impurity is used as a start raw material, and the nature catalyst support of porosity silicon carbide of the honeycomb structure calcinated and acquired by the configuration of a request of this after shaping and desiccation in a 1600-2200-degree C temperature requirement is indicated by JP,6-182228,A.
[0004] A vitrification material adds to the fireproof constituent which, on the other hand, contains an easy-oxidizable material or an easy-oxidizable material in JP,61-26550,A, and the silicon-carbide Plastic solid with which the manufacture approach of the vitrification material content refractories characterized by to carry out nakedness baking of the Plastic solid mixed, kneaded, and fabricated and fabricated in the furnace of a non-oxidizing atmosphere adds and fabricates an organic binder and the inorganic binder of a clay mineral system, textile glass yarn, and a silicic-acid lithium system to silicon-carbide powder at JP,8-165171,A is indicated with binding material, respectively.
[0005] Moreover, after adding and fabricating binding material, such as glassiness flux or argillaceous, to the carbonization system elementary particle used as the aggregate as the manufacture approach of the conventional nature sintered compact of porosity silicon carbide, the method of burning, hardening and manufacturing the Plastic solid at the temperature which said binding material fuses is also introduced to said JP,6-182228,A.
[0006] furthermore, to JP,61-13845,B and JP,61-13846,B Silica sand, a pottery grinding object, aluminum2O3, TiO2, the metallic oxide of ZrO2 grade, The fireproof particle by which the particle size regulation was carried out to the predetermined grain size which consists of silicon carbide, a nitride, boride, or other fireproof ingredients The suitable fireproof particle pitch diameter, fireproof particle particle size distribution, tube-like object porosity, tube-like object average pore size, tube-like object pore volume, tube-like object septum thickness, etc. are indicated about the high-temperature-service ceramic filter formed in the porous cylinder-like-object-with-base-like object with fireproof binding material, such as water glass, a frit, and a cover coat.
[0007] In addition, in JP,8-13706,B, the manufacture approach of the silicon carbide / metal silicon complex which has the structure which it comes to join to one through metal silicon, and said complex using the silicon carbide and metal silicon which heat-treated silicon accumulation biomass under an

argon or nitrogen-gas-atmosphere mind, and were formed is indicated.
[0008]
[Problem(s) to be Solved by the Invention] Although a silicon carbide component evaporates from a silicon carbide particle front face, the neck section grows because this condenses in the contact section between particles (neck section), and an integrated state is obtained with the sintering gestalt (necking) by the recrystallization reaction of the silicon carbide powder itself shown in said JP,6-182228,A In order for this to have to cause cost quantity since a very high burning temperature is required, and to evaporate silicon carbide and to have to carry out elevated-temperature baking of the ingredient with a high coefficient of thermal expansion, there was a problem that a baking yield fell.
[0009] Moreover, it had the fault that growth of the neck section will be barred since the sintering-machine style concerned stops fully functioning, and will originate in this, and the reinforcement of a filter will fall by sintering by the recrystallization reaction of the above silicon carbide powder itself if it is going to manufacture the filter which is high porosity, and the filter which has 50% or more of especially porosity.
[0010] Furthermore, though advantageous, a catalyst is support in that the above-mentioned ingredient has thermal conductivity very as high as 30 or more W/mK, and local generation of heat is suppress, for example, and when a particulate is use for the filter of the method which oxidizes and burns and is reproduce continuously, time amount is take for the temperature of support to go up very much according to the description of there be little particulate alimentation and be easy to radiate heat. Therefore, in order to take time amount for temperature to go up to the temperature as which a catalyst functions, the particulate cinder arose and it also had problems, like regeneration efficiency falls.
[0011] The technique of combining the coal-for-coke-making-ized silicon powder shown in JP,61-26550,A or JP,6-182228,A by glassiness Although it is low and ends with 1000-1400 degrees C as a burning temperature for example, in using as an ingredient of the diesel particulate filter (DPF) for removing the particulate contained in the exhaust gas discharged from a diesel power plant in the sintered compact produced by this technique When it was going to burn the particulate which uptake was carried out to the filter and deposited for filter playback, since thermal conductivity was small, there was a trouble that local generation of heat arose.
[0012] Furthermore, although the filter shown in JP,61-13845,B and JP,61-13846,B was porosity, septa are 5-20mm and a thick cylinder-like-object-with-base-like object, and were not able to apply it to the bottom of a high SV (space velocity) condition like the filter for motor exhaust purification.
[0013] Moreover, although the complex concerned could also be made into porosity by the complex shown in JP,8-13706,B, and its manufacture approach, it was not easy to face to use it as a filter and to secure sufficient porosity, and it was difficult to use it as a filter for carrying out uptake removal of the particulate matter contained in dust-containing fluid like diesel-power-plant exhaust gas especially in the complex concerned.
[0014] thermal conductivity be set as the moderate numeric value , and this invention be fully porosity and high specific surface area , and aim at offer the honeycomb structure object which can be suitably use also under high SV conditions as a filter for motor exhaust purification by processing of ****** , catalyst support , etc. , and its manufacture approach while being able to manufacture them cheaply with comparatively low burning temperature , though it be make in view of such a conventional situation and a fireproof particle like a silicon carbide particle be include .
[0015]
[Means for Solving the Problem] According to this invention, it is the honeycomb structure object which has the circulation hole of a large number penetrated to the shaft orientations divided by the septum, and honeycomb structure object ** characterized by being porosity is offered including the fireproof particle and metal silicon used as the aggregate.
[0016] Moreover, after according to this invention adding metal silicon and an organic binder in a fireproof particle raw material, fabricating the plastic matter mixed, and kneaded and obtained in a honeycomb configuration, carrying out temporary quenching of the acquired Plastic solid and removing the organic binder in a Plastic solid, manufacture approach ** of the honeycomb structure object

characterized by carrying out actual baking is offered.

[0017]

[Embodiment of the Invention] Since metal silicon for the honeycomb structure object of this invention to combine these refractoriness particle with a fireproof particle as aforementioned is included, it can be made to sinter with a comparatively low burning temperature at the time of the manufacture, and the yield can be raised while holding down a manufacturing cost. Moreover, even if it burns the particulate deposited for filter playback when it is used, for example for DPF since it has high thermal conductivity as compared with the conventional structure which used glassiness for association of a fireproof particle by having used metal silicon for association of a fireproof particle, local generation of heat which damages a filter does not arise. Furthermore, since this invention is not the cylinder-like-object-with-base-like object of a thick wall as shown in JP,61-13845,B or JP,61-13846,B but a porous honeycomb structure object, it can be used under high SV conditions as a filter, catalyst support, etc. for motor exhaust purification.

[0018] Moreover, as for the honeycomb structure object of this invention, it is desirable that the fireproof particle which constitutes the honeycomb structure object concerned has the structure combined with metal silicon in a part of the particle front face. It is the honeycomb structure object applied to this invention at drawing 2 , and the microphotography which is the crystal structure of the nature sintered compact of silicon carbide is shown. For a white part, metal silicon 10 and a gray part are [the silicon carbide particle 11 and a black part] pores 12 among drawing. Thus, it turns out that the particles to which the silicon carbide particle 11 which is a fireproof particle exists in a perimeter in a part of the particle front face are combined with metal silicon 10. In addition, the manufacture approach of the nature sintered compact of silicon carbide shown in drawing 2 is mentioned later.

[0019] Since the above-mentioned structure is formed without using the metal silicon beyond the need, it can suppress the eburnation by fusion of the metal silicon comrade who occurs in the process of baking. For this reason, sufficient porosity is secured although the pressure loss at the time of using as a filter is suppressed low. Furthermore, since it has the high heat conductivity even if it burns the particulate deposited for filter playback while the high porosity in the case of using as DPF for carrying out uptake removal of the particulate contained in the exhaust gas discharged from a diesel power plant since it also has the high heat conductivity therefore etc. is fully secured, local generation of heat which a filter damages does not arise.

[0020] As for the honeycomb structure object of this invention, it is desirable that the viewpoint which avoids the above local generation of heat to the thermal conductivity is 5 or more W/mK.

[0021] Moreover, as for the honeycomb structure object of this invention, it is desirable to have the structure combined with metal silicon as the microstructure after the fireproof particle had stopped the raw material particle shape. When using the particulate matter contained in dust-containing fluid in the honeycomb structure object of this invention as a filter for carrying out uptake removal, it is desirable to make the porosity into 30 - 90% of range. If the porosity of a honeycomb structure object runs short of filtration velocity at less than 30% and exceeds 90%, the reinforcement as the structure runs short. Furthermore, when using for the application for which we are anxious about the pressure loss of the filter for motor exhaust purification etc., it is desirable to make porosity into 40% or more.

[0022] furthermore, in being the honeycomb structure object used as filters which must suppress pressure loss low, such as a filter of the method which a catalyst is supported [method] and burns a particulate continuously It is desirable that there is porosity and thermal conductivity is in the range of 5 - 30 W/mK 50 to 90%, it is still more desirable that there is porosity and thermal conductivity is in the range of 7 - 28 W/mK 50 to 80%, and especially the thing that there is porosity and thermal conductivity is in the range of 9 - 25 W/mK 53 to 70% is desirable.

[0023] In the honeycomb structure object used as a filter of the method which makes a catalyst support, since pressure loss goes up by supporting a catalyst, it is necessary to set up porosity highly beforehand. Therefore, at less than 50%, since pressure loss becomes large with the filter of this method, porosity is not desirable. On the other hand, if porosity exceeds 90%, since the reinforcement as the structure runs short, it is not desirable.

[0024] Furthermore, in the honeycomb structure object used as a filter of said method, it is necessary to suppress that local stress occurs in a filter according to generating of the uneven temperature distribution by local generation of heat. Therefore, it becomes difficult for thermal conductivity to suppress local generation of heat effectively in less than 5 W/mK. Since it may originate in that the effectiveness of heat dissipation is large, that there is little particulate alimentation, etc. on the other hand if the heat conductivity exceeds 30 W/mK, a particulate cinder may arise while taking great time amount to carry out a temperature up even to the temperature as which a catalyst functions that it is hard to go up temperature, and the regeneration efficiency of a filter may fall, it is not desirable.
[0025] In addition, although the catalyst supported by the filter as used in the field of this invention is a catalyst used for the purpose of particulate oxidation combustion and decomposition of NOX and oxides, such as noble metals, such as platinum, palladium, a rhodium, iridium, and silver, or an alumina, a zirconia, a titania, Seria, and an iron oxide, etc. can specifically be used, this invention is not limited to these things.
[0026] When using the honeycomb structure object of this invention as a filter similarly, as for the average pore size of a honeycomb structure object, determining according to the object to filter is desirable. For example, when using as DPF for carrying out uptake removal of the particulate contained in the exhaust gas discharged from a diesel power plant, it is desirable to make average pore size into the range of 2-50 micrometers. if a pressure loss goes up remarkably and it exceeds 50 micrometers conversely also by little deposition of a particulate [pore size / average] in less than 2 micrometers -- particulate base -- since an omission happens, it is not desirable.
[0027] Although the suitable content of the metal silicon in the honeycomb structure object of this invention changes also with the particle size and particle shape of a fireproof particle, it is desirable to consider as 5 - 50% of the weight of within the limits to the total quantity of a fireproof particle and metal silicon, and it is still more desirable to consider as 15 - 40% of the weight of within the limits. Less than 5 % of the weight of association by the metal silicon of the fireproof particles which adjoin since binding material is insufficient is insufficient, and it becomes difficult to obtain the reinforcement which thermal conductivity not only falls, but can maintain the structure of a thin wall like honeycomb structure. Conversely, if it exceeds 50 % of the weight, in the point which originates in metal silicon existing more than it can combine fireproof particles appropriately, and a honeycomb structure object (sintered compact) contracts too much by sintering, and evils, such as a porosity fall and average pore size contraction, concur with, it is not desirable.
[0028] As for the thickness of the septum with which the circulation hole (cel) of a honeycomb structure object is divided, it is desirable to be referred to as 4 or more (102 micrometers or more) mil. Under 4mil (102 micrometers) of the reinforcement as the structure is [the thickness of a septum] insufficient. Moreover, reinforcement had porosity and a close relation, when in the case of the honeycomb structure object of this invention setting up the thickness of a septum so that the thickness and the porosity of a septum might fill the following relation, required reinforcement was obtained, and the desirable thing became clear.
[Equation 4] Thickness (micrometer) >= porosity (%) x4[0029] of a septum Furthermore, if the thickness of a septum is set up so that the thickness and the porosity of a septum may fill the following relation, since sufficient reinforcement will be obtained, it is more desirable.
[Equation 5] Thickness (micrometer) >= porosity (%) x5[0030] of a septum On the other hand, when using as filters, such as DPF, it is desirable to set thickness of a septum to 50 or less (1270 micrometers or less) mil. When the thickness of a septum exceeds 50mil (1270 micrometers), it is for being anxious about the lack of filtration velocity, or a pressure drop buildup. In addition, there is close relation to porosity also about this, and a problem can be avoided by setting up the thickness of a septum so that the thickness and the porosity of a septum may fill the following relation.
[Equation 6] Thickness (micrometer) <= porosity (%) x20[0031] of a septum As for the cel consistency of a honeycomb structure object, it is desirable to consider as the range of 5-1000 cel / square inch (0.7 - 155 cel / cm2). a cel consistency -- 5 cels / square -- under in an inch (0.7 cels / cm2), while becoming insufficient [reinforcement] as a honeycomb structure object, when it uses as a filter, filtration areas

also run short. On the contrary, it is not desirable in order to cause a pressure drop buildup, if 1000 cel / square inch (155 cels / cm2) is exceeded.

[0032] Next, the manufacture approach of the honeycomb structure object of this invention is explained. In manufacturing the honeycomb structure object of this invention, first, metal silicon and an organic binder are added in a fireproof particle raw material, it mixes and kneads in it, and the plastic matter for shaping is obtained.

[0033] although especially the class of fireproof particle to be used is not limited -- an oxide system -- aluminum2 -- O3, ZrO2, Y2O3, and a carbide system -- SiC and a nitride system -- Si3 -- thermal resistance of SiC is high for applications, such as DPF which particles, such as N4, AlN, and other mullites, are used suitably, for example, is often exposed to an elevated temperature at the time of combustion processing of an are recording particulate, and it is suitably used for them. In addition, although there is a case containing the impurity of minute amounts, such as Fe, aluminum, and calcium, in the raw material used for fireproof particle metallurgy group silicon, you may use it as it is and what performed and refined chemical processing of chemical washing etc. may be used.

[0034] As for the mean particle diameter of a fireproof particle raw material, it is desirable that it is 2 to 4 times the average pore size of the honeycomb structure object (sintered compact) finally acquired by this manufacture approach. Since the honeycomb structure object acquired by this manufacture approach has a comparatively low burning temperature, the particle shape and particle size of a fireproof particle raw material are maintained in general until after baking. Therefore, such reinforcement high [particle size is too small to desired pore size in said ratio being under 2 double, and] cannot be obtained that it will be combined long and slender with metal silicon, and a fireproof small particle group will form big pore and can maintain the structure of a thin wall like a honeycomb structure object as a result.

[0035] Moreover, for example, the recrystallization SiC conventionally applied to the porosity honeycomb structure object when a fireproof particle is a SiC particle The SiC particle combined from the reaction mechanism with metal silicon like the honeycomb structure object of this invention to needing an aggregate raw material particle size almost equivalent to the pore size considered as a request Since **** [particle size / twice / more than / the pore size], when it is going to obtain the same pore size, compared with Recrystallization SiC, a cheap raw material can be used coarsely, and a cost merit is also large.

[0036] On the contrary, when said ratio exceeds 4 times, the particle size of the fireproof particle used to desired pore size is too large, and it is not desirable by being densely filled up with a fireproof particle in the phase of shaping at the point which becomes difficult [it / to obtain desired pore] for the gap, and causes a porosity fall for a filter application further.

[0037] Metal silicon melts during baking, wets the front face of a fireproof particle, and bears the role which combines particles. Although the suitable addition changes also with the particle size and particle shape of a fireproof particle, it is desirable to make it become to the total quantity of a fireproof particle and metal silicon 5 - 50% of the weight of within the limits. At less than 5 % of the weight, since metal silicon exists superfluously more than it can combine fireproof particles appropriately if binding material is insufficient, and the reinforcement which can maintain the structure of a thin wall like honeycomb structure cannot be obtained but it exceeds 50 % of the weight conversely, evils, such as a porosity fall and average pore size contraction, occur at the same time.

[0038] As for the mean particle diameter of metal silicon, it is desirable that it is 50% or less of the mean particle diameter of the fireproof particle which is the aggregate. If the particle size exceeds 50% of the particle size of a fireproof particle in order to move melting and gathering by baking so that it may coil around a fireproof particle, at the time of shaping, the space which this metal silicon particle occupied serves as a big opening, metal silicon remains, a fall on the strength is caused, or when using it as a filter, it will cause filter degradation (leakage in filtration).

[0039] Moreover, also in order for the direction which generally mixes two or more sorts of raw material powder with a grain-size difference at the time of extrusion molding of a honeycomb structure object to be able to extrude smoothly and to obtain an organization suitable as a porous body, it is desirable to make mean particle diameter of metal silicon into 50% or less of the mean particle diameter of the

fireproof particle which is the aggregate.

[0040] In order to use a fireproof particle as the aggregate and to carry out extrusion molding of the plastic matter which comes to blend an ostomy agent etc. metal silicon and if needed to a honeycomb configuration smoothly, it is desirable to add one or more sorts of organic binders 2% of the weight or more by outside ** as a shaping assistant to the total quantity of the main raw material (a fireproof particle raw material and metal silicon). However, in order that the addition exceeding 30 % of the weight may cause superfluous high porosity and may make the lack of on the strength result after temporary quenching, it is not desirable.

[0041] Furthermore, when the thickness of a septum carries out extrusion molding to the honeycomb structure object below 20mil (508 micrometers), it is desirable to add in 4 - 20% of the weight of the range. an addition -- less than 4 % of the weight -- ** -- if it is difficult to extrude in a thin wall [like] and it exceeds 20 % of the weight conversely, it will become difficult to maintain the configuration after extrusion.

[0042] When using a honeycomb structure object as a filter, an ostomy agent may be added at the time of preparation of a plastic matter in order to raise porosity. As for the addition of an ostomy agent, it is desirable to consider as 30 or less % of the weight by outside ** to the total quantity of the main raw material (a fireproof particle raw material and metal silicon). If an addition exceeds 30 % of the weight, porosity will become high too much and it will result in the lack of on the strength.

[0043] In addition, when acquiring the honeycomb structure object which is 50% or more of high porosity, it is desirable to add an ostomy agent. The honeycomb structure object which is the high porosity by which pore volume distribution was controlled is producible by choosing suitably the class of ostomy agent used at this time, mean particle diameter, etc. That is, although the gap between the particles of the fireproof particle which is the aggregate serves as pore in this invention, the honeycomb structure object of high porosity which has the pore volume distribution which consists of two pore volume distribution of the gap between the particles of a fireproof particle and the remains of destruction by fire of an ostomy agent is producible by carrying out suitable amount addition of the ostomy agent which has mean particle diameter 1.2 to 4 times the particle size of the fireproof particle which is the aggregate. Therefore, the flexible materials design corresponding to required pore volume distribution becomes possible by choosing suitably the particle size of a fireproof particle and an ostomy agent.

[0044] On the other hand, in order that pore size may produce a large honeycomb structure object, when using fireproof particle metallurgy group silicon with a big particle size, a plastic matter can be smoothly extruded at the time of extrusion molding by carrying out suitable amount addition of the ostomy agent which has 0.5 or less times [of the mean particle diameter of a fireproof particle] particle size. Therefore, the honeycomb structure object of high porosity can be produced, without lowering a moldability.

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the honeycomb structure object used for a filter, catalyst support, etc. for motor exhaust purification.

[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1] The honeycomb structure object characterized by being porosity including the fireproof particle and metal silicon which are the honeycomb structure object which has the circulation hole of a large number penetrated to the shaft orientations divided by the septum, and serve as the aggregate.
[Claim 2] The honeycomb structure object according to claim 1 with which said fireproof particle has the structure combined with said metal silicon in a part of the fireproof particle front face.
[Claim 3] The honeycomb structure object according to claim 1 whose thermal conductivity is 5 or more W/mK.
[Claim 4] The honeycomb structure object according to claim 1 which has the structure combined with said metal silicon after said fireproof particle had stopped the raw material particle shape.
[Claim 5] The honeycomb structure object according to claim 1 said whose fireproof particle is a silicon carbide particle.
[Claim 6] The honeycomb structure object according to claim 1 used as a filter which carries out uptake removal of the particulate matter contained in dust-containing fluid.
[Claim 7] The honeycomb structure object according to claim 1 in the range whose porosity is 30 - 90%.

[Claim 8] The honeycomb structure object according to claim 1 in the range whose average pore size is 2-50 micrometers.
[Claim 9] The honeycomb structure object according to claim 2 which has thermal conductivity in the range of 5 - 30 W/mK while porosity is in the range which is 50 - 90%.
[Claim 10] The honeycomb structure object according to claim 1 whose content of said metal silicon is 5 - 50% of the weight of the range to the total quantity of said fireproof particle raw material and metal silicon.
[Claim 11] The honeycomb structure object according to claim 1 whose thickness of said septum is 102-1270 micrometers.
[Claim 12] The honeycomb structure object according to claim 1 with which the thickness of said septum and the porosity of a honeycomb structure object fill the following relation.
[Equation 1] thickness (micrometer) >= porosity (%) x4 of a septum -- [Claim 13] The honeycomb structure object according to claim 1 with which the thickness of said septum and the porosity of a honeycomb structure object fill the following relation.
[Equation 2] thickness (micrometer) >= porosity (%) x5 of a septum -- [Claim 14] The honeycomb structure object according to claim 1 with which the thickness of said septum and the porosity of a honeycomb structure object fill the following relation.
[Equation 3] thickness (micrometer) <= porosity (%) x20 of a septum -- [Claim 15] The honeycomb structure object according to claim 1 whose cel consistencies are 0.7 - 155 cel / cm2.
[Claim 16] The manufacture approach of the honeycomb structure object characterized by carrying out actual baking after adding metal silicon and an organic binder in a fireproof particle raw material, fabricating the plastic matter mixed, and kneaded and obtained in a honeycomb configuration, carrying

out temporary quenching of [redacted] acquired Plastic solid and removing the organic binder in a Plastic solid.

[Claim 17] The manufacture approach according to claim 16 that said fireproof particle raw material is a silicon carbide particle raw material.
[Claim 18] The manufacture approach according to claim 16 that the mean particle diameter of said fireproof particle raw material is 2 to 4 times the average pore size of the honeycomb structure object finally acquired.
[Claim 19] The manufacture approach according to claim 16 that the addition of said metal silicon is 5 - 50% of the weight of the range to the total quantity of said fireproof particle raw material and metal silicon.
[Claim 20] The manufacture approach according to claim 16 that the mean particle diameter of said metal silicon is 50% or less of the mean particle diameter of the fireproof particle which is the aggregate.
[Claim 21] The manufacture approach according to claim 16 which adds said organic binder in 2 - 30% of the weight of the range by outside ** to the total quantity of said fireproof particle raw material and metal silicon.
[Claim 22] The manufacture approach according to claim 16 which adds an ostomy agent in 30 or less % of the weight of the range by outside ** to the total quantity of said fireproof raw material particle and metal silicon in case said plastic matter is prepared.
[Claim 23] The manufacture approach according to claim 16 of carrying out temporary quenching of said Plastic solid at temperature lower than the temperature which said metal silicon fuses.
[Claim 24] The manufacture approach according to claim 16 of carrying out said this baking in a 1400-1800-degree C temperature requirement.

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PRIOR ART

[Description of the Prior Art] The porous honeycomb structure object is widely used as catalyst support for supporting the catalyst component which purifies the filter for carrying out uptake removal of the particulate matter contained in dust-containing fluid like diesel-power-plant exhaust gas, or the harmful matter in exhaust gas. Moreover, using a fireproof particle like a silicon carbide (SiC) particle as a component of such a honeycomb structure object is known.
[0003] As a concrete related technique, while having a predetermined specific surface area, the silicon carbide powder containing an impurity is used as a start raw material, and the nature catalyst support of porosity silicon carbide of the honeycomb structure calcinated and acquired by the configuration of a request of this after shaping and desiccation in a 1600-2200-degree C temperature requirement is indicated by JP,6-182228,A.
[0004] A vitrification material adds to the fireproof constituent which, on the other hand, contains an easy-oxidizable material or an easy-oxidizable material in JP,61-26550,A, and the silicon-carbide Plastic solid with which the manufacture approach of the vitrification material content refractories characterized by to carry out nakedness baking of the Plastic solid mixed, kneaded, and fabricated and fabricated in the furnace of a non-oxidizing atmosphere adds and fabricates an organic binder and the inorganic binder of a clay mineral system, textile glass yarn, and a silicic-acid lithium system to silicon-carbide powder at JP,8-165171,A is indicated with binding material, respectively.
[0005] Moreover, after adding and fabricating binding material, such as glassiness flux or argillaceous, to the carbonization system elementary particle used as the aggregate as the manufacture approach of the conventional nature sintered compact of porosity silicon carbide, the method of burning, hardening and manufacturing the Plastic solid at the temperature which said binding material fuses is also introduced to said JP,6-182228,A.
[0006] furthermore, to JP,61-13845,B and JP,61-13846,B Silica sand, a pottery grinding object, aluminum2O3, TiO2, the metallic oxide of ZrO2 grade, The fireproof particle by which the particle size regulation was carried out to the predetermined grain size which consists of silicon carbide, a nitride, boride, or other fireproof ingredients The suitable fireproof particle pitch diameter, fireproof particle particle size distribution, tube-like object porosity, tube-like object average pore size, tube-like object pore volume, tube-like object septum thickness, etc. are indicated about the high-temperature-service ceramic filter formed in the porous cylinder-like-object-with-base-like object with fireproof binding material, such as water glass, a frit, and a cover coat.
[0007] In addition, in JP,8-13706,B, the manufacture approach of the silicon carbide / metal silicon complex which has the structure which it comes to join to one through metal silicon, and said complex using the silicon carbide and metal silicon which heat-treated silicon accumulation biomass under an argon or nitrogen-gas-atmosphere mind, and were formed is indicated.

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EFFECT OF THE INVENTION

[Effect of the Invention] As explained above, though fireproof particles, such as a silicon carbide particle, are included, since it can be made to sinter with a comparatively low burning temperature at the time of the manufacture, the honeycomb structure object of this invention can improve [yield's] while holding down a manufacturing cost, and can be offered cheaply. Moreover, even if it burns the particulate deposited for filter playback when it is used, for example for DPF since it has high thermal conductivity as compared with the conventional structure which combined the fireproof particle using glassiness, local generation of heat which damages a filter does not arise. Furthermore, since porosity and thermal conductivity are predetermined numerical range and are the honeycomb structure object of the low porosity of pressure loss, it can be suitably used also under high SV conditions as a filter for motor exhaust purification which made the catalyst support.

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TECHNICAL FIELD

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Although a silicon carbide component evaporates from a silicon carbide particle front face, the neck section grows because this condenses in the contact section between particles (neck section), and an integrated state is obtained with the sintering gestalt (necking) by the recrystallization reaction of the silicon carbide powder itself shown in said JP,6-182228,A In order for this to have to cause cost quantity since a very high burning temperature is required, and to evaporate silicon carbide and to have to carry out elevated-temperature baking of the ingredient with a high coefficient of thermal expansion, there was a problem that a baking yield fell.
[0009] Moreover, it had the fault that growth of the neck section will be barred since the sintering-machine style concerned stops fully functioning, and will originate in this, and the reinforcement of a filter will fall by sintering by the recrystallization reaction of the above silicon carbide powder itself if it is going to manufacture the filter which is high porosity, and the filter which has 50% or more of especially porosity.
[0010] Furthermore, though advantageous, a catalyst is support in that the above-mentioned ingredient has thermal conductivity very as high as 30 or more W/mK, and local generation of heat is suppress, for example, and when a particulate is use for the filter of the method which oxidizes and burns and is reproduce continuously, time amount is take for the temperature of support to go up very much according to the description of there be little particulate alimentation and be easy to radiate heat. Therefore, in order to take time amount for temperature to go up to the temperature as which a catalyst functions, the particulate cinder arose and it also had problems, like regeneration efficiency falls.
[0011] The technique of combining the coal-for-coke-making-ized silicon powder shown in JP,61-26550,A or JP,6-182228,A by glassiness Although it is low and ends with 1000-1400 degrees C as a burning temperature

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MEANS

[Means for Solving the Problem] According to this invention, it is the honeycomb structure object which has the circulation hole of a large number penetrated to the shaft orientations divided by the septum, and honeycomb structure object ** characterized by being porosity is offered including the fireproof particle and metal silicon used as the aggregate.
[0016] Moreover, after according to this invention adding metal silicon and an organic binder in a fireproof particle raw material, fabricating the plastic matter mixed, and kneaded and obtained in a honeycomb configuration, carrying out temporary quenching of the acquired Plastic solid and removing the organic binder in a Plastic solid, manufacture approach ** of the honeycomb structure object characterized by carrying out actual baking is offered.
[0017]
[Embodiment of the Invention] Since metal silicon for the honeycomb structure object of this invention to combine these refractoriness particle with a fireproof particle as aforementioned is included, it can be made to sinter with a comparatively low burning temperature at the time of the manufacture, and the yield can be raised while holding down a manufacturing cost. Moreover, even if it burns the particulate deposited for filter playback when it is used, for example for DPF since it has high thermal conductivity as compared with the conventional structure which used glassiness for association of a fireproof particle by having used metal silicon for association of a fireproof particle, local generation of heat which damages a filter does not arise. Furthermore, since this invention is not the cylinder-like-object-with-base-like object of a thick wall as shown in JP,61-13845,B or JP,61-13846,B but a porous honeycomb structure object, it can be used under high SV conditions as a filter, catalyst support, etc. for motor exhaust purification.
[0018] Moreover, as for the honeycomb structure object of this invention, it is desirable that the fireproof particle which constitutes the honeycomb structure object concerned has the structure combined with metal silicon in a part of the particle front face. It is the honeycomb structure object applied to this invention at drawing 2 , and the microphotography which is the crystal structure of the nature sintered compact of silicon carbide is shown. For a white part, metal silicon 10 and a gray part are [the silicon carbide particle 11 and a black part] pores 12 among drawing. Thus, it turns out that the particles to which the silicon carbide particle 11 which is a fireproof particle exists in a perimeter in a part of the particle front face are combined with metal silicon 10. In addition, the manufacture approach of the nature sintered compact of silicon carbide shown in drawing 2 is mentioned later.
[0019] Since the above-mentioned structure is formed without using the metal silicon beyond the need, it can suppress the eburnation by fusion of the metal silicon comrade who occurs in the process of baking. For this reason, sufficient porosity is secured although the pressure loss at the time of using as a filter is suppressed low. Furthermore, since it has the high heat conductivity even if it burns the particulate deposited for filter playback while the high porosity in the case of using as DPF for carrying out uptake removal of the particulate contained in the exhaust gas discharged from a diesel power plant since it also has the high heat conductivity therefore etc. is fully secured, local generation of heat which a filter damages does not arise.

[0020] As for the honeycomb structure object of this invention, it is desirable that the viewpoint which avoids the above local generation of heat to the thermal conductivity is 5 or more W/mK.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the graph which plotted porosity, reinforcement (MPa), and thermal conductivity (W/mK)(%) to the amount (wt%) of the blended metal Si powder.
[Drawing 2] It is the microphotography which is the crystal structure of the nature sintered compact of silicon carbide produced in the example 1.
[Description of Notations]
10 -- Metal silicon, 11 -- A silicon carbide particle, 12 -- Pore.

[Translation done.]

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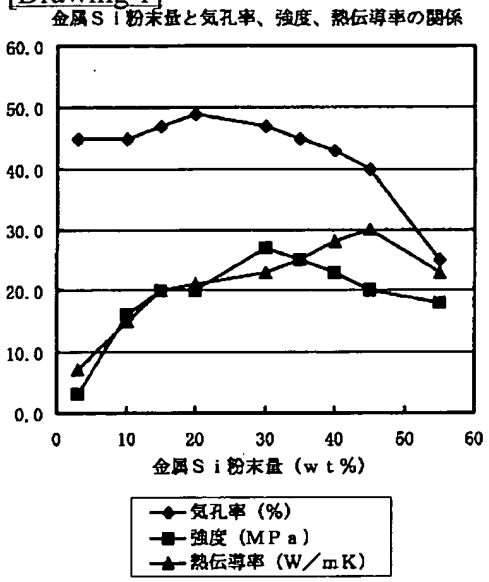
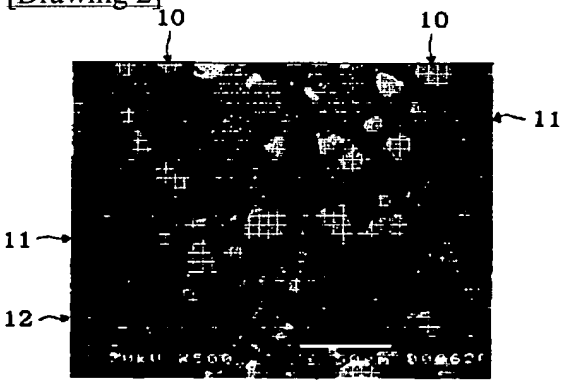
DRAWINGS

[Drawing 1]



[Drawing 2]



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